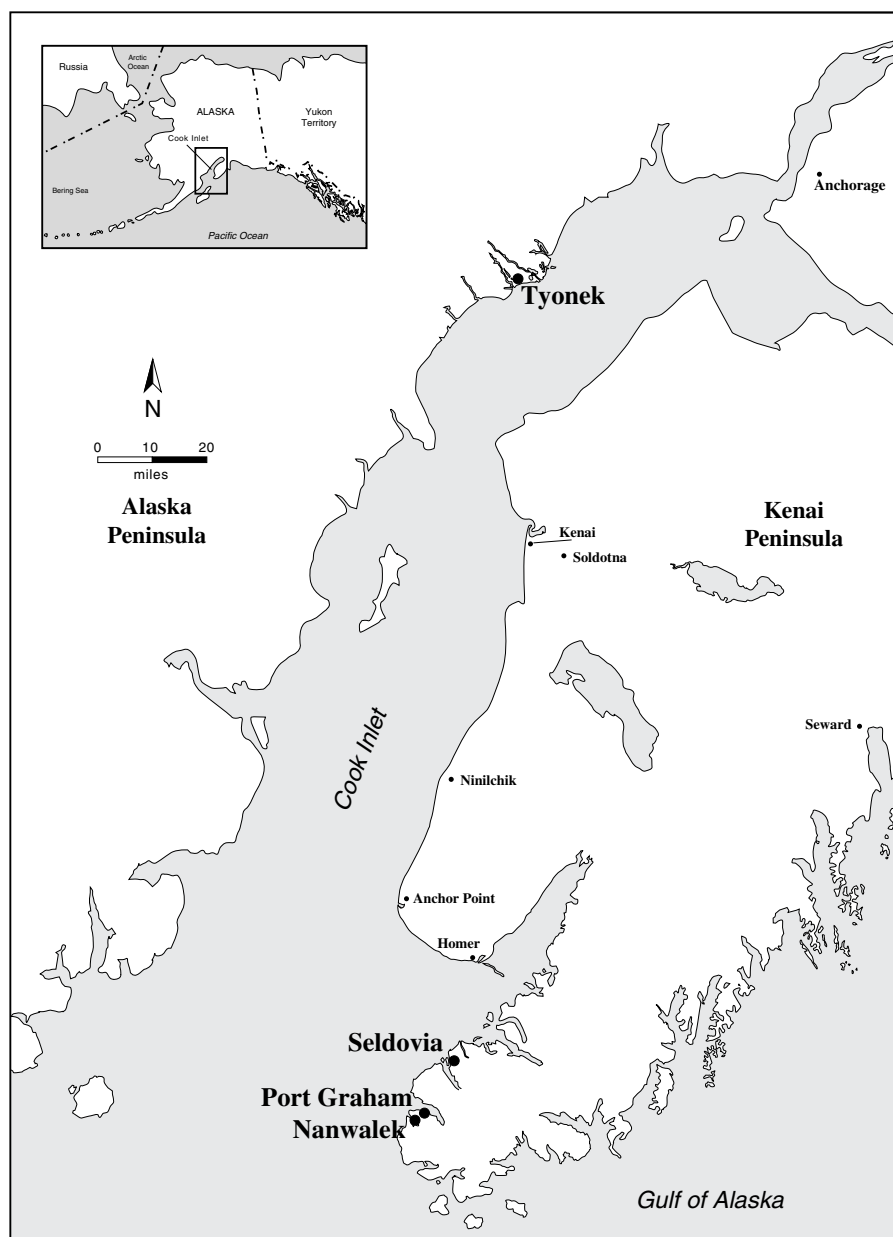




# Appendix K

## Audit Report: Samples preparation of Cook Inlet samples by Axys Analytical Services Sidney, B. C. Canada



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**AUDIT REPORT**

**SAMPLE PREPARTION OF COOK INLET SAMPLES**

**AXYS ANALYTICAL SERVICES**

**SIDNEY, B.C. , CANADA**

prepared by

Ecology and Environment, Inc.

Analytical Services Center

On March 2, 1998, operations pertinent to the preparation and compositing of samples collected at Cook Inlet, performed under AR#2084, were observed by Ecology and Environment, Inc. (E&E). This work is being performed as a subcontract under EPA Megalab Contract #68-C6-0004. Evaluation was limited to the preparation of samples; homogenization and compositing. Analytical procedures for analysis were not reviewed as part of this audit.

## **SAMPLE STORAGE**

Samples have been stored frozen at the Axys facility since they were received in June, 1997. Two large walk-in freezers have been utilized for storage of the samples. Samples are stored at -20 C. The freezers are equipped with an electronic alarm system which sounds should temperatures climb above -5C. These freezers are located just outside the laboratory building. A key is required to gain entry to the freezers.

Samples were received frozen in large cooler chests. Each chest was placed immediately in the walk-in freezer. Sample log-in and chain of custody verification was performed at a later time, one cooler at a time. No problems with freezer storage have been noted since the beginning of this project.

With the exception of the long term archival freezer chest, the temperature of all refrigerators and freezers is recorded each business day. A chart for each month is posted on each appliance. Control limits and corrective actions (notify supervisor) are indicated on each sheet. Completed monthly records are filed by the Sample Custodian.

The homogenization and compositing process generates many equipment rinsates (proofs) which are stored refrigerated until analysis.

## **SAMPLE CUSTODY**

Sample Custody was neat and orderly. Record keeping appears to be appropriate. The Sample Custodian is responsible for tracking and archiving temperatures on all refrigerators and freezers. SOPs are available in the work area. The deep freeze archival storage temperatures are checked biweekly, rather than daily. All other areas are checked daily.

Package receipt logs are completed upon sample receipt. Cooler temperatures are checked and sample condition.

Chain of custody forms are checked versus samples received. Any discrepancies are noted, and the Project Chemist is notified. Val Scott is the Project Chemist assigned to this project.

All standards are stored separately, away from samples.

## **SAMPLE PREPARATION**

A composite of red salmon was being prepared during the observation period. Each individual fish to be included in the composite was frozen and appeared to have been frozen immediately after catch. The color and condition of the fish were excellent, there were no signs of any deterioration. The identity of fish to be composited has been relayed from the Sample Control Center to Axys Project Chemist, Val Scott, who in turn has relayed this information to Sample Custody and Sample Preparation Supervisor. The correct samples are pulled, their identities confirmed and they are assigned the EPA number.

Each fish was weighed and its weight recorded in a laboratory notebook under its assigned EPA number. Each fish was chopped into sizes appropriate to fit the grinder. A 3 horse power stainless steel meat grinder was utilized for the salmon. Other smaller appliances are available for other sample types such as clams or vegetation. All appliances are cleaned and covered with aluminum foil when not in use.

Entire fish are ground, including scales, heads and organs. The five fish included in this composite were each passed through the grinder and collected into a large stainless steel bowl. The bowl was quartered and opposite quarters were blended well and then passed through the grinder a second time. The second passings were then quartered, opposite quarters blended and all passed through the grinder a third time.

Noticeable changes in consistency and color were observed between the three grindings. The third grinding appeared extremely consistent, no variations in texture or color were noticeable.

The sample was aliquoted into the designated, labeled containers.

To keep the number of rinsate required for this project to a minimum, samples are being processed in an order to only utilize one piece of homogenizing equipment per day as much as possible.

Technicians performing the sample preparation, homogenizing and compositing were familiar with the SOP and performed all functions accordingly.

## **SUMMARY**

Axys Analytical Services is a neat and competent operation. The staff is knowledgeable and familiar with the tasks assigned. SOPs are readily available in each work area.

The only negatives observed were facility related. The laboratory is housed in a separate building, away from the business and administrative areas. The large freezers are located outside the buildings. Although secure, due to the possibility of inclement weather and the decreased likelihood of discovering a problem, it would be better if the freezers were inside.

The sample preparation area was rather confining, but the area was clean and organized. There are plans underway to enlarge this area.